

Screen Test for Cadmium and Nickel Plates

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Abstract

A new procedure is described here which was recently developed to quantify loading uniformity of nickel and cadmium plates and to screen finished electrodes prior to cell assembly. The technique utilizes the initial solubility rates of the active material in a standard chemical deloading solution at fixed conditions. The method can provide a reproducible indication of plate loading uniformity in situations where high surface loading limits the free flow of deloading solution into the internal porosity of the sinter plate. A preliminary study indicates that "good" cell performance is associated with higher deloading rates.

I. Introduction:

The performance of nickel cadmium (Ni-Cd) and nickel hydrogen (Ni-H₂) battery cells has been found to be critically dependent on the variability in active material loading of the nickel and cadmium electrodes used in them. Several recent ground test problems and orbital battery anomalies have been attributed to such inappropriate loading of active material, which is often caused by process instability, or a lack of manufacturing process control. At the present time, no effective procedure is available to quantify the loading uniformity at the microscope level, or to screen the finished electrodes prior to cell assembly. The new technique described here shows correlation between plate performance and deloading rates of cadmium and nickel plates. This report communicates the preliminary results obtained from the new method.

II. Experimental:

Experiments were performed to develop a procedure for characterization of loading uniformity of cadmium and nickel plates. Two small samples (approximately 1.85 cm x 1.85 cm) of finished plates were chosen for analysis from each plate, with one sample being within the middle of the electrode and away from the edges of the electrodes, and the other sample being near one edge. The edges of the samples were coated with viscous epoxy to prevent the leaching

of the active material from the cut edges of the samples. The epoxy-edged samples were allowed to cure at room temperature for approximately 24 hours. The weights with epoxy and dimensions of samples were obtained. During the epoxy coating process, small spots on the samples were occasionally smeared with wet epoxy and therefore, an area correction factor was measured to determine the actual surface areas of samples. A standardized chemical extraction solution was prepared by dissolving 154g of reagent grade ammonium acetate in 1.3 liter (ℓ) of concentrated ammonium hydroxide (reagent grade) and then diluting to 2.0ℓ with deionized water. Active material in the sample electrodes was allowed to leach out by complexing with the ammonia solution, held at $50^{\circ}\text{C} \pm 2^{\circ}\text{C}$, for a period of 5 minutes, after which the samples were thoroughly rinsed until the rinse solution was near to a neutral pH condition. After drying at room temperature for about 12 hours or more, the samples were then weighed, and the weight losses were recorded to determine the standard deloading rate at 50°C . Care must be taken to maintain uniform solution temperature ($50^{\circ}\text{C} \pm 2^{\circ}\text{C}$) during the deloading. A sample holder was used to keep the specimen in a vertical position. Pure nickel is suggested as an ideal sample holder material due to its inert nature in the basic ammonia solution.

III. Results and Discussion:

Method Development:

Cadmium electrodes were obtained from "bad" and "good" cells, and subjected to the previous described procedure to determine the appropriate extraction conditions which could provide useful data representative of surface loading characteristics. "Bad" electrodes, as defined here, exhibited anomalously rapid cadmium migration in cells, causing premature short circuits in some cases. It is expected that any surface loading which blocks movement of the deloading solution into the interior of the porous plate should reduce the deloading rate. Figure 1 indicates the kinetic data obtained from two different cell lots, L6A, and L2A which are known to be "good" and "bad", respectively. The preliminary data indicates that the initial solubility rate of active material in the standard extraction solution for fixed conditions provides reproducible measurements of surface loading uniformity of cadmium plates. The five minutes extraction time was selected as the standard time interval to measure the initial deloading rate of cadmium in ammonia solution, because this interval gave roughly 10-20% deloading.

Results:

Figure 2 shows preliminary data, which indicate that "good" cell performance is typically associated with higher deloading rates, ranging from 6-10 mg/(min.cm²). This implies that the interior structure of the electrode is more open to chemical deloading and thus there is lesser degree of blockage within the porous structure of the electrodes. Samples from lot 2A, 2B, 3, and 4 are identified with "bad" electrodes, which are usually characterized by lower deloading rates, i.e. 0.3-3 mg/(min.cm²).

Figure 3 shows the variation in deloading rates for electrode samples from different spirals within one production post. These differences could be attributed to the variation of the spiral locations in the impregnation tank, resulting in higher or lower surface active material loading. These results clearly show that variability can be significant not only from lot to lot but also within a single post of production.

The screen test has also been applicable to the nickel electrode, for which excessive amounts of surface loading of active material has often resulted in poor electrode utilization and accelerated swelling over cycle life. As shown in Figure 4, the deloading rates of nickel plate samples were found to be proportional to the fractional utilization which is defined as the ratio of the C/2 plate capacity to 0.0 V (vs. Hg/HgO) over the total capacity stored in the electrode. It is believed that with an appropriate data base, an expression of electrode utilization as a function of various parameters (EQ. 1) could be obtained and used to screen finished plates prior to cell assembly, thus preventing costly problems from "bad" plates in spacecraft batteries or in ground tests.

$$\text{Utilization} = k_1L + k_2P + k_3W + k_4Co + k_5D + k_6 \quad (\text{EQ. 1})$$

Where

L : Loading level

P: Sinter porosity

W: Wt. of sinter

Co: Percentage of cobalt in active material

D: deloading rate, related to surface loading uniformity

k₁, k₂, k₃, k₄, k₅, k₆ are constants

L, P, W, and Co could be found by chemical analysis of electrodes, and D may be determined from the deloading experiment.

IV. Summary and Recommendations:

Currently, we are in the process of perfecting the technique and obtaining more data to evaluate method reliability and effectiveness under various conditions. Communication with battery manufacturers will be considered to obtain representative electrodes that are known to be either "good" or "bad" to build up a large data base in an attempt to predict electrode utilization as a function of multiple variables which affect plate performance. In the near future this technique, the methods of interpretation, and our results will be forwarded to spacecraft battery manufacturers to allow and encourage them to utilize this new capability.

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Kinetic Data

Figure 1

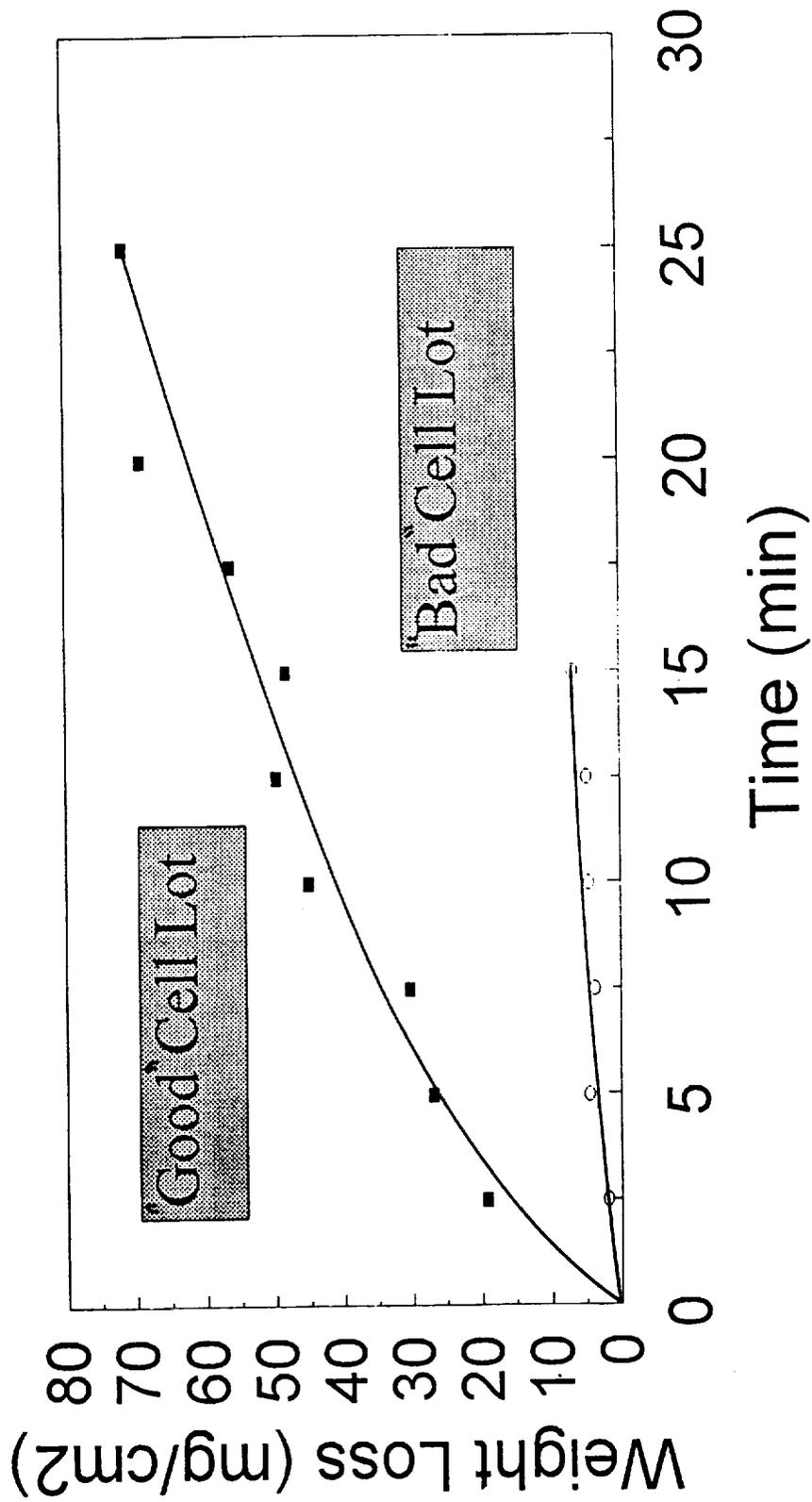


Figure 2

Deloading Experiment for Cd Plates

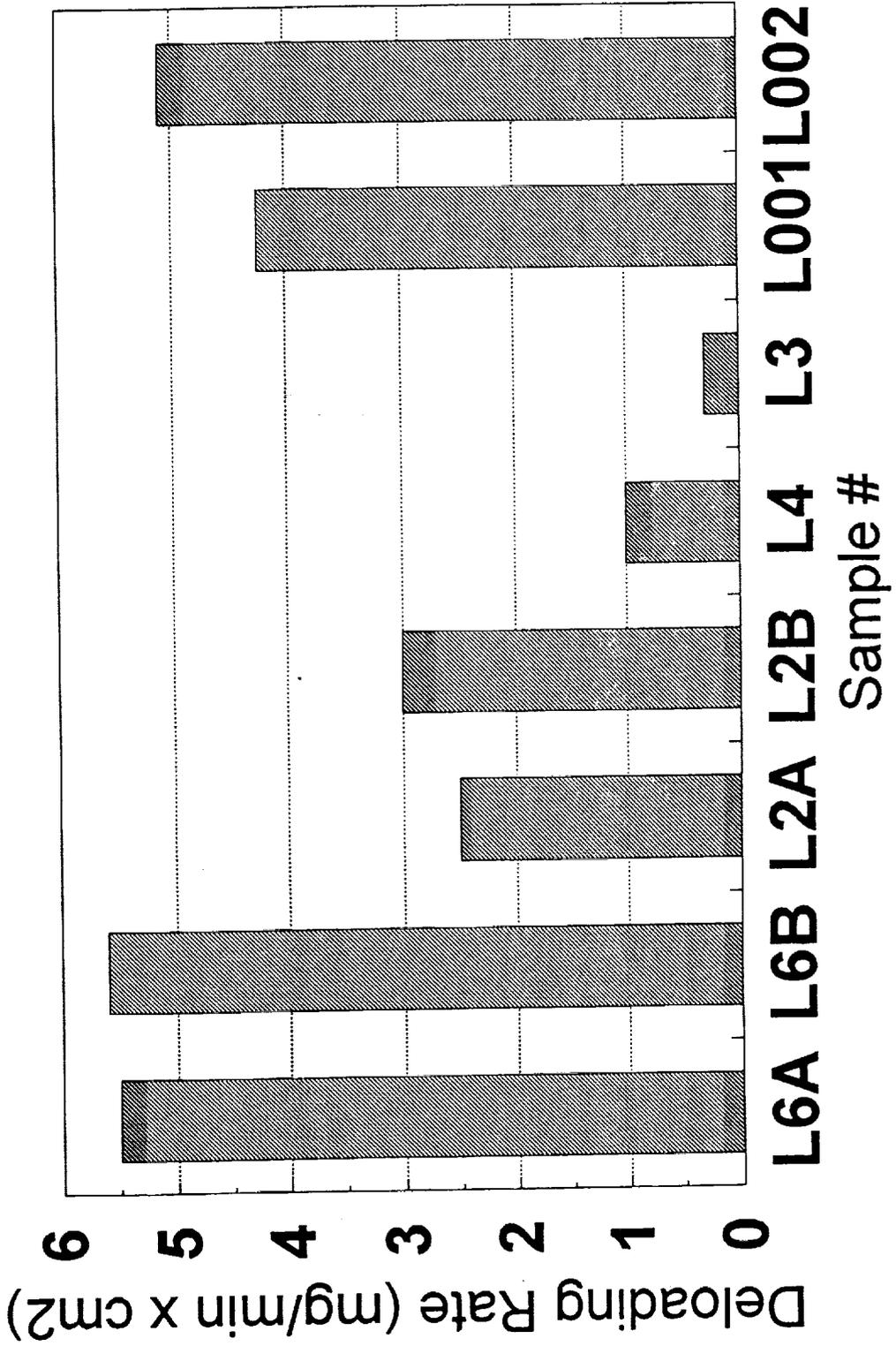


Figure 3

Deloading Exp. for Cadmium Plates Lot 1B

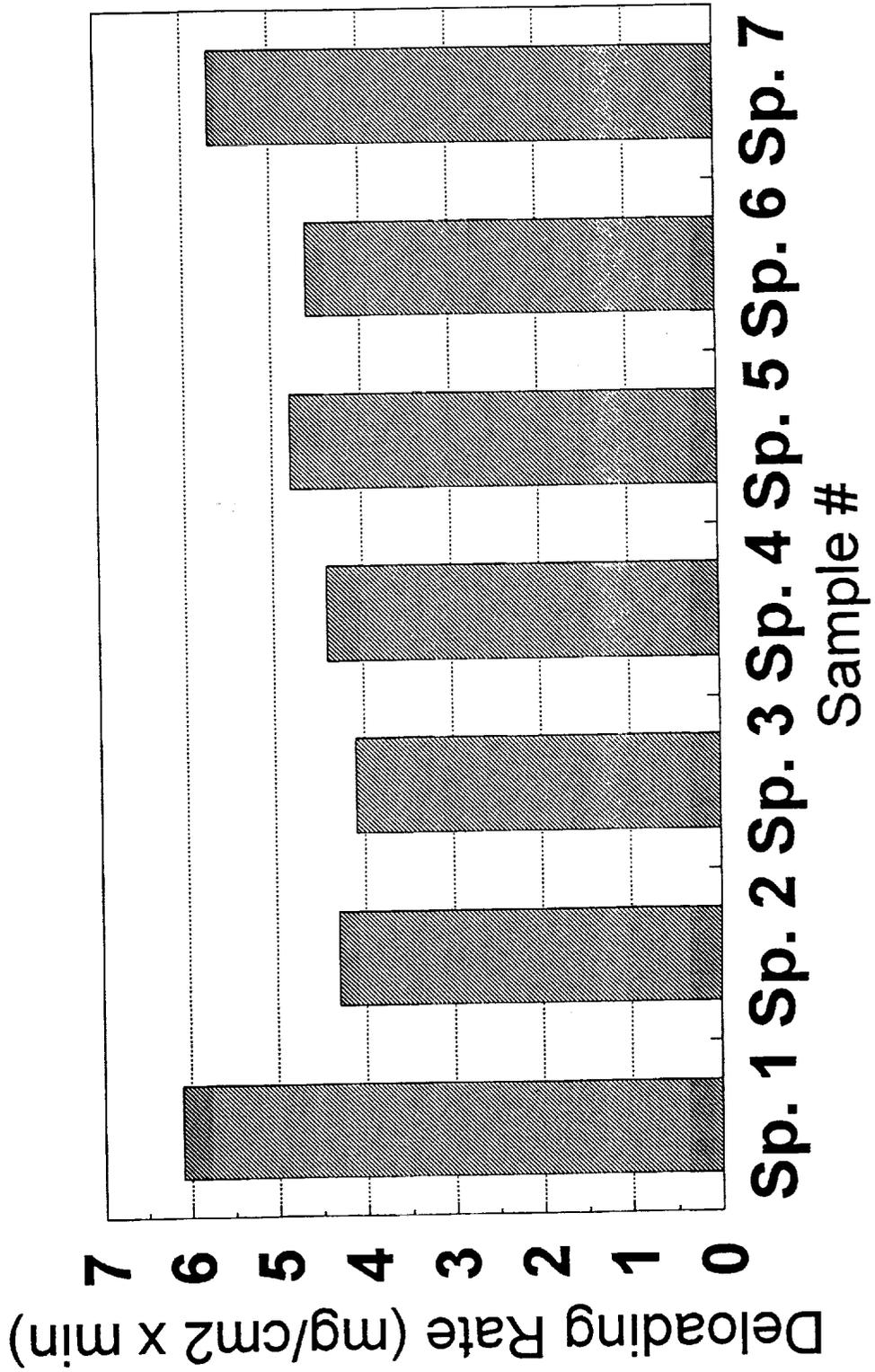


Figure 4

Correlation Between Deloading Rate of Ni Plates and Fractional Utilization

